FINAL REPORT

Introducing Interfacial Bond Failure at the Elastomerprimer Interface Using Tailored Infrared-Sonication Hybrid Technology

SERDP Project WP18-1347

AUGUST 2019

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

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1. REPORT DATE *(DD-MM-YYYY)*
08/22/2019

2. REPORT TYPE
SERDP Final Report

3. DATES COVERED *(From - To)*
8/23/2018 - 8/22/2019

4. TITLE AND SUBTITLE
Introducing Interfacial Bond Failure at the Elastomerprimer Interface Using Tailored Infrared-Sonication Hybrid Technology

5a. CONTRACT NUMBER
18-C-0049

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Jaspreet S. Dhau, Molekule Incorporation

Terence Parker, Florida Polytechnic University, Lakeland

5d. PROJECT NUMBER
WP18-1347

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Molekule Incorporation: 1184 Harrison St., San Francisco, CA 94103

Florida Polytechnic University, Lakeland: Lakeland, FL 33608

8. PERFORMING ORGANIZATION REPORT NUMBER
WP18-1347

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Strategic Environmental Research and Development Program
4800 Mark Center Drive, Suite 17D03
Alexandria, VA 22350-3605

10. SPONSOR/MONITOR'S ACRONYM(S)
SERDP

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
WP18-1347

12. DISTRIBUTION/AVAILABILITY STATEMENT
DISTRIBUTION STATEMENT A. Approved for public release: distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT
The objective of this project, SERDP WP18-C2-1347, was to develop an innovative and cost-effective coating removal technique for removing thick elastomeric coatings from Department of Defense Weapon Systems. The developed technology aims to eliminate the need for chemical- or media-based treatments, thus reducing environmental problems. This was achieved through an innovative approach involving the use of tailored infrared and ultrasonication treatments of the coated substrates. The effect of different operating parameters such as time of irradiation, surface temperature, sonication power/intensity were investigated to determine the optimal conditions for coating removal without damaging the underlying substrate or causing environmental problems.

15. SUBJECT TERMS
Longwave Infrared Radiation, Mediumwave Infrared Radiation, Shortwave Infrared Radiation, Fourier transform infrared, spectroscopy, Differential scanning calorimetry,

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Jaspreet S. Dhau
UNCLASS	UNCLASS	UNCLASS	UNCLASS	44	19b. TELEPHONE NUMBER *(Include area code)* 813-451-5081

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ACKNOWLEDGEMENTS

Molekule Inc. would like to acknowledge the support given by Florida Polytechnic University, Air Force Research Laboratory (AFRL), Naval Air (NAVAIR) research laboratory and Radiant Energy Systems. Their assistance and help to make this project a success is greatly appreciated.

ABSTRACT

Objective: The objective of this project, SERDP WP18-C2-1347, was to develop an innovative and cost-effective coating removal technique for removing thick elastomeric coatings from Department of Defense Weapon Systems. The developed technology aims to eliminate the need for chemical- or media-based treatments, thus reducing environmental problems. This was achieved through an innovative approach involving the use of tailored infrared and ultrasonication treatments of the coated substrates. The effect of different operating parameters such as time of irradiation, surface temperature, sonication power/intensity were investigated to determine the optimal conditions for coating removal without damaging the underlying substrate or causing environmental problems.

Technical Approach: Our approach was based on the concept of introducing bond failure at the surface coat-substrate interface by selective absorption of IR and/or impinging of the topcoat with the use of ultrasonication technique. The degraded interfacial adhesion strength then allowed the specialty coating to be completely removed with the application of a pull at a certain angle to the substrate-coating interface. The development of tailored IR-Sonication coating removal technology was achieved through the following tasks and subtasks.

Task 1: Characterization of the surface coat and the underlying substrate coat

ST 1.1. FTIR and Specular reflectance in the range of 2.5 μm to 22.5 μm for the surface coat polyurethane-filler composite.

ST 1.1.1. FTIR and Specular reflectance in the range of 2.5 μm to 22.5 μm for the underlying substrates (epoxy composite, bismaleimide composite, aluminum and titanium).

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Task 3: Fabrication and Testing of IR-Sonication probe

ST 3.1. Design and Fabrication of IR-Sonication probe based on results from tasks 1 & 2.

ST 3.2. Testing of IR-Sonication probe and generation of data.

Task 4: Characterization of surface coat and underlying substrates after treatment from task3, economic Analysis and Final Technical Report

ST 4.1. Characterization of Surface coat after treatment.

ST 4.2. Costing and Economic Analysis.

ST 4.3. Quarterly Progress Report.

ST 4.4. Final technical report.

Results: Optimum ultrasonication frequency/amplitude and infrared spectral range, where the surface coat showed the maximum absorption and the underlying substrate the minimum absorption, were determined. Based on these results, tailored IR and sonication treatments were applied on several different specialty coating samples provided by AFRL and NAVAIR. All the tasks were completed, and milestones were achieved as mentioned in the project plan. One of the key deliverables, i.e., introducing interfacial bond failure between the topcoat and the underlying substrate, was accomplished with a significantly better result than that was originally proposed in the proposal (25-50% bond strength reduction, **Milestone 2.1 and Final Deliverable 2**). On an average, a 65-75% peel-strength reduction was achieved by the use of tailored IR treatment alone and IR-Ultrasonic combination. Both, the shortwave infrared (SW-IR) and mediumwave infrared (MW-IR) treatments demonstrated peel-strength reductions in the range of 75-50% with varied exposure time. The desired peel-strength reduction was achieved significantly quicker with SW-IR than that achieved with MW-IR exposure.

Two approaches were used for ultrasonication experiments; 1) directly applying ultrasonic energy to the top layer of the sample, 2) applying sonication while maintaining a very thin layer of water between the ultrasonic horn and the top layer. First approach was most successful in removing thick elastomeric sealants from the NAVAIR samples. The second approach was adopted for the IR-sonication hybrid treatment. The latter procedure showed a higher peel strength reduction than that achieved with the application of IR alone.

Cost analysis was carried out using the average energy usage of each technology. To remove one square feet of coating using IR treatments, the energy required was in the range of 100-200 kJ/ft^2. This is accounted to $0.001-0.004 /ft^2 for an electricity tariff of $0.065/kWh. Ultrasonic approach gives energy consumption of 200-300kJ/ft^2 and would account to $0.002-0.006/ft^2. This approach is an economically viable option considering the basic operation cost of the technology. Further studies are needed to investigate the complicated shapes and the initial machine fabrication costs.

In order to determine any adverse effect on the underlying substrates, thermogravimetric analysis (TGA), FTIR and SEM imaging of the treated and as-received samples were investigated. The results established that IR and ultrasonication exposure had no detrimental effect on the physical and chemical makeup of the underlying substrates. These results not only provide a solid proof-of-concept of the proposed technology but also provide optimized parameters to successfully implement this technology on an industrial scale.

Expected Benefits: The developed coating removal technology is an environmental-friendly method as it does not produce any toxic gas or suspended solid waste in the air. Operation cost for this technology is very low compared to the conventional coating removal technologies. Coating removal is achieved at a much lower temperature than some of the energy-intensive technologies such as laser ablation. We have successfully addressed SERDP's goal by developing a large-scale non-chemical, non-media removal process for thick, elastomeric coatings and treatments on Department of Defense Weapon Systems.

1 INTRODUCTION

Molekule Inc. has led a one-year Strategic Environmental Research and Development Program (SERDP) limited scope project WP18-C2-1347 entitled, “Introducing interfacial bond failure at the elastomer-primer interface using tailored infrared-sonication hybrid technology.” The aim was to develop an infrared-sonication based technology to remove thick elastomeric coatings from aircraft and Department of Defense (DOD) weapon systems. The final report presents the outcomes on this one-year project. The report explains the activities that were carried out during the given one-year period; the technical results, discussions, conclusion and implications for the future research/implementation of an infrared and ultrasonication based coating removal system on a large-scale area specialty application.

1.1 Objective

The limited-scope project was aimed at addressing the ubiquitous issue of removal of thick, elastomeric coatings and treatments without damaging the underlying substrate or causing environmental problems while being ergonomically less challenging. The idea was to use tailored IR, sonication and IR-sonication hybrid procedures to introduce interfacial bond failure at the interface of the elastomeric coating and underlying substrate. The objective of this proposals was to acquire data demonstrating proof-of-concept of the proposed technology and identify parameters for its successful implementation on an industrial scale.

2 BACKGROUND

Most of the DOD weapon systems and associated support equipment are coated in some form to provide substrates with protection from corrosion and weathering. While traditional primers, paints and topcoats are used for many applications, there are some specialty applications where thicker and more elastomeric coatings are used for special properties. These applications would include rain erosion coatings, fuel tank coatings and specialty coatings. Each of these coating systems present a particular challenge. Rain erosion coating is often well cross-linked but still relatively thin compared, for example, to fuel tank coating. DOD specialty coatings are usually filled with a metallic or ceramic particulate, and fuel tank coating is often very thick and highly cross-linked to withstand the environment of its intended use.

Within routine maintenance cycles, coatings frequently have to be removed, either locally for repairs, or totally for refurbishment. A variety of coating removal techniques have been developed including laser, microwave, flash-lamps, ice blasting, cryogenic and CO_2 blasting, plastic media blasting, wheat starch or similar blasting, and chemical strippers [1 – 3]. Many of these, especially the media blasting methods and the chemical stripping, create a waste stream that is much more than just the removed coating. In addition to the coating, it can include the blasting media, the transport fluid, or the waste solvent stream from chemical stripping. Chemical stripping, in particular, has the potential to create a hazardous material waste stream as well as volatile organic compounds (VOCs) and/or hazardous air pollutants (HAPs).

The specialty coatings of interest here are relatively difficult to remove by virtue of their inherent chemical resistance and/or elastomeric nature. Chemical stripping can be difficult because the diffusion-controlled nature of the chemical penetration makes removal of a thick coating protracted [4, 5]. The highly elastomeric nature of the thicker films renders media blasting difficult because the media tends to just bounce off of the film rather than erode as expected. Laser ablation of surface coatings can cause heat damage to the substrate. If the incident photon energy is applied for too long a period of time, significant heat can transfer to the substrate, leading to the potential for substantial damage [6, 7]. Additionally, media blasting can have a deleterious effect on the substrate [8, 9].

Consequently, these specialty coatings are often removed through some combination of chemical stripping and manual scraping. An improved method of specialty coating removal would have the potential to improve both environmental compliance through reduced waste stream and worker health and safety through reduced exposure to hazardous materials and reduced manual effort.

2.1 Technical approach

Our approach was based on the concept of introducing interfacial bonding failure at the surface coat-substrate interface by selective absorption of IR radiation emitted from an IR source with simultaneous impinging of area using an ultrasonication probe to induce cavitational wear (Fig. 1). The degraded interfacial adhesion strength then allows the specialty coating to be completely removed with the application of a pull at a certain angle to the substrate-coating interface, which can be achieved manually or by a fully automatic process.

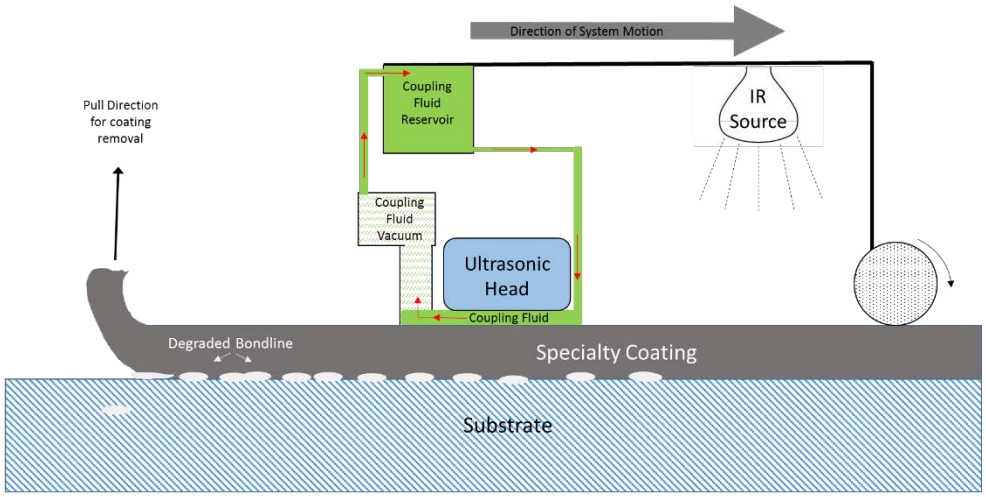

Fig. 1. Schematic of application of IR-Sonication hybrid technology to remove specialty coatings

2.2 Infrared Radiation Treatment

IR radiant heating is used extensively in industry, particularly for drying of materials or fusing of coatings. Due to its radiative heat transfer mode, IR heating has distinct advantages over other forms of heating; it can be selectively and readily absorbed by the material or tailored potions of the material. Infrared heating is applied to the part surface by direct transmission from an emitter. A part of the energy emitted is reflected off the surface, the other absorbed by the substrate and some transmitted though the substrate. The absorption characteristics depend on the type of material, the color, and the surface finish.

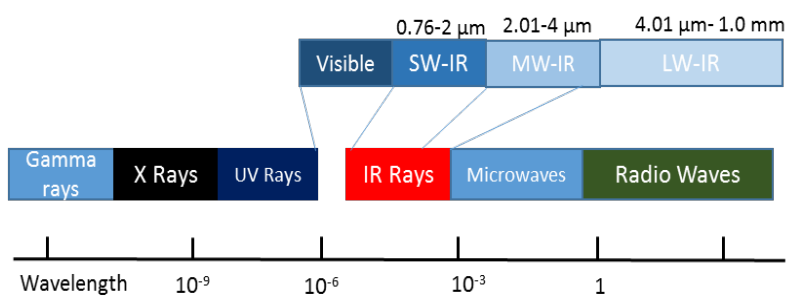

Fig. 2. IR Spectrum

The behavior of IR energy depends on the wavelength, the distance between the substrate and the emitter, the mass of the part, the surface area and the color of the substrate. It is well established that shorter wavelength infrared radiation penetrates further into the substrate but is more sensitive to changes in the color of the substrate and the medium IR range is well absorbed by polymers. We have explored the full IR spectral range in order to find an optimum IR spectral wavelength range where the surface coat shows the maximum emissivity and the underlying area minimum emissivity.

2.3 Ultrasonication Approach

In addition to IR, we have also studied the application of ultrasonication for complete or partial removal of thick elastomeric coatings. We have used sonication probe to induce cavitation wear in the surface coating. This was done in a way to accelerate the chemical and physical wear induced at the interface and surface of the substrate. In the present work, we have studied the effect of different operating parameters such as time of irradiation, and sonication power/intensity in the coating removal operation to optimize operating parameters. We have studied the effect of sonication alone on the as-received specimens, and also in combination with IR exposure.

2.4 Characterization and Testing of Treated Substrates

Various characterization techniques and testing standards were used to validate the success of the developed technology. A Standard Test Methods for Rubber Property—Adhesion to Rigid Substrates (ASTM D429) method B, 90° stripping test was used to quantify the peel-strength of the top coat and the underlying substrate [14]. Fourier transform infrared spectroscopy (FTIR) was used to identify the IR absorption range and determine the chemical makeup of the surfaces of treated and untreated substrates. Differential scanning calorimetry (DSC), Thermogravimetric analysis (TGA) and Scanning electron microscope (SEM) were used to investigate the structural integrity of the underlying substrate.

3 TECHNICAL ACTIVITIES ACCOMPLISHED

Development and evaluation of the IR and ultrasonication approaches for coating removal will be discussed in this section. The proposed tasks are discussed in detail with the results accomplished in the following text.

3.1 Task 1: Characterization of the surface coat and the underlying substrate coat

3.1.1 Sample Preparation

One of the key tasks of the project was to obtain uniformly coated aluminum and composite samples that have strong adhesion between the top coat and the underlying substrate. Aluminum substrates were coated in-house either by using a dip coating method or a spray-painting technique. Al Coupons of 1x2 inch were cut and roughened with a sand paper to improve adhesion with the polyurethane coating. This was followed by a dip or spray coat strep (Fig. 3). It was found that the spray coating gave a more uniform coating of polyurethane film than that achieved with the dip coating method.

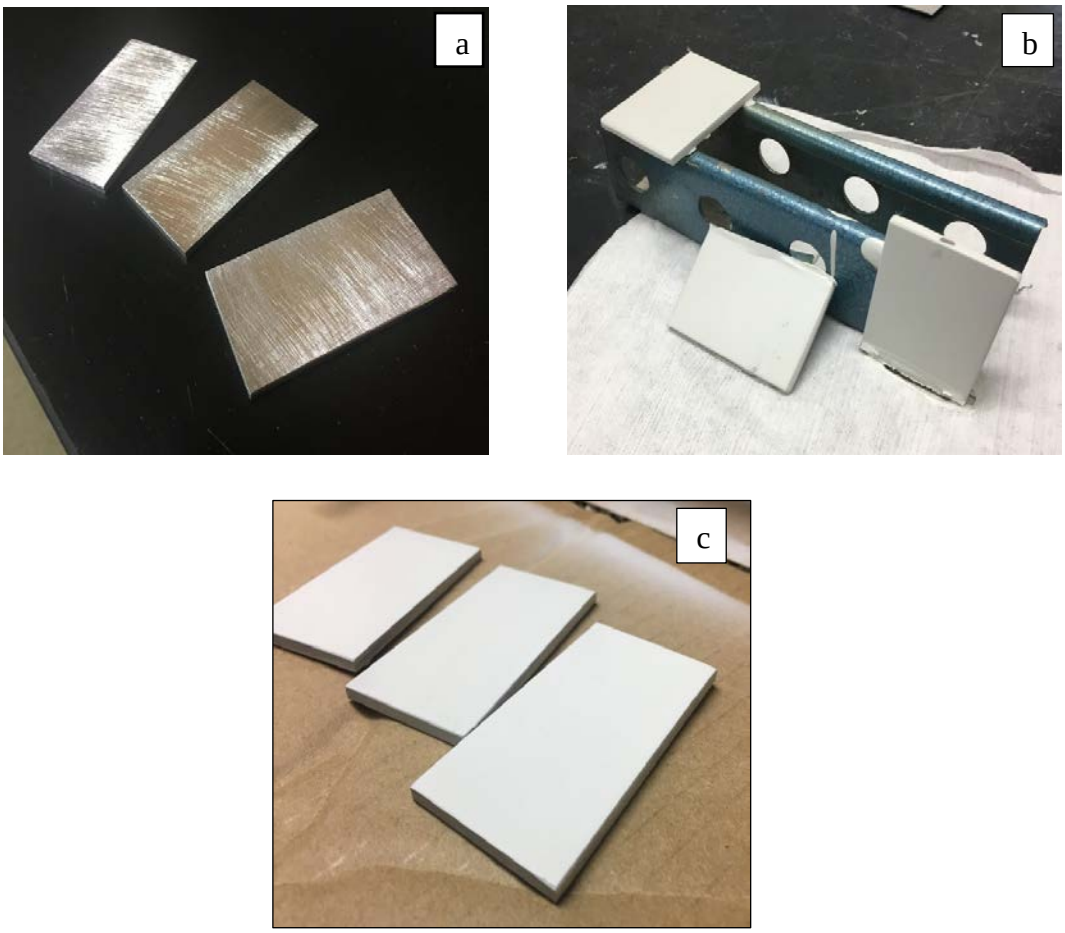

Fig. 3. In-house coated aluminum substrates; a) roughened Al substrate, b) Dip coated (Prime and top coat both), c) spray painted (both primer and top coat)

3.1.2 Air Force Research Lab (AFRL) Samples

In order to test the proposed technology and optimize the operation parameters on real defense materials, samples with specialty coatings were received from Air Force Research Lab AFRL and NAVAIR. Eight different sets of samples (6” x 6”) were received from AFRL and tested (Fig. 4a). Description of the test samples has been provided below:

1. AFRL-RE* coating on aluminum substrate: Coating thickness, 0.5 mm
2. AFRL-RE coating on aluminum substrate: Coating thickness, 1 mm
3. AFRL-RE coating on composite substrate: Coating thickness, 0.5 mm
4. AFRL-RE coating on composite substrate: Coating thickness, 1 mm
5. AFRL-FP* coating on aluminum substrate: Coating thickness, 0.5 mm
6. AFRL-FP coating on aluminum substrate: Coating thickness, 1 mm
7. AFRL-FP coating on composite substrate: Coating thickness, 0.5 mm
8. AFRL-FP coating on composite substrate: Coating thickness, 1 mm

   *DOD Specialty Coating

We also got sealant samples from NAVAIR (Fig. 4b) that are used in fuel tanks. The samples had sealant coating with irregular surface thickness and appearance. After receiving these samples from AFRL and NAVAIR, small rectangular coupons of 3”x 0.5” dimension were cut and machined (Fig. 5).

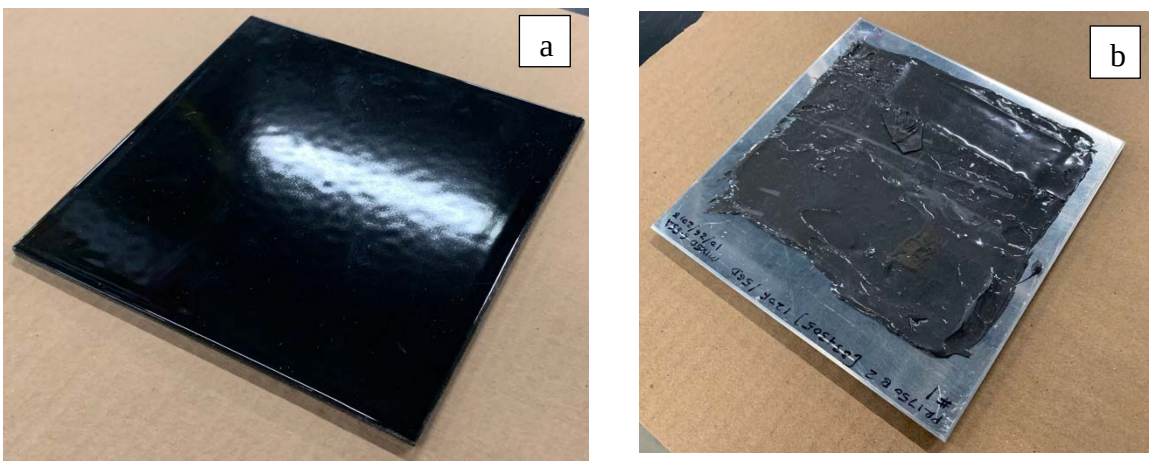

Fig. 4.a) AFRL coated sample with DOD specialty coating, b) NAVAIR coated sealant sample

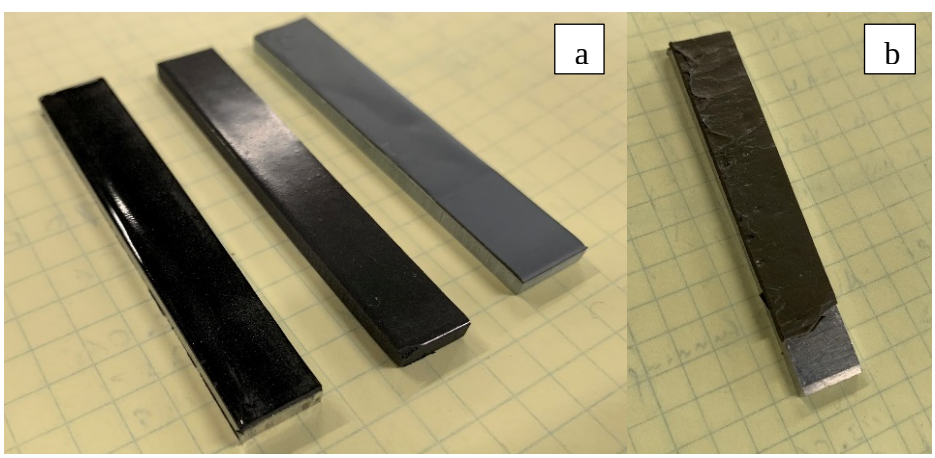

Fig. 5.Samples from a) AFRL and b) NAVAIR

3.1.3 Optimum IR spectral range

During the course of the present study, efforts were made to assess optimum IR spectral range, where the surface coat showed the maximum absorption and the underlying substrate the minimum absorption. The task was performed on in-house coated samples (dip and spray coated) and AFRL samples and accomplished by using Jasco FT/IR-6300 spectrometer with an attached Emission Optical System and a TGS detector. We found that the surface coat polyurethane-filler materials show complete attenuation of IR radiation in the SW and a part of MW, however it showed some transmittance in the 3.4 µm to 5.7 µm range. It was also found that the underlying aluminum substrate showed no absorption in this range. There is a clear difference in IR range absorption of polyurethane coatings when compared with aluminum (Fig. 6) and composite substrates (refer Appendix, Fig. A2). In-house spray and the dip coated samples as well as the samples received from AFRL had common absorption ranges. Even though there is a slightly high absorption range in the long wave length, due to its low energy density, the amount of heat absorption is limited. Short exposure times were preferred to reduce the heat transfer through conduction from top layer to the underlining substrate, therefore, MW-IR (medium-wave infrared) and SW-IR (short-wave infrared) were selected. The SW-IR wave length range is around 0.7 µm to 3 µm which was just around lower limit of the IR emitter unit designed and fabricated in collaboration with Radiant Energy Systems Inc., our Industrial Partner on this project. We also used IR Emitter Cassette Module in the Fast-response Medium wave (2.0 µm), and Medium wave (2.5-3.0 µm) range.

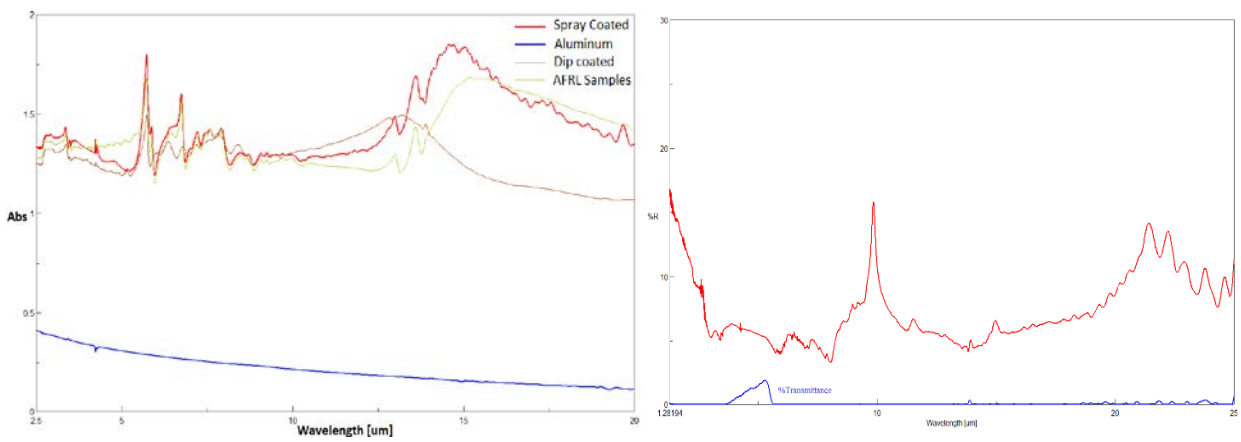

Fig. 6. a) % Absorption of IR spectra of coated and uncoated substrates, b) %Reflectance and %Transmission of the rain erosion coating material

The IR experimental unit that we tested had both the Shortwave (SW) IR and Mediumwave (MW) IR heaters but had the option to run it separately and at different power levels (Fig. 7 and 8).

Fig. 7. IR emitter unit with SW-IR and MW-IR modules

Fig. 8. IR emitter unit (interchangeable cassette) with control panel

Differential Scanning Calorimeter (DSC) analysis was conducted on the samples to determine the degradation temperature and oxidative stability of the top coat and the underlying substrate. As evident from the Fig. 9, the elastomer coated sample degrades at 315 ^{o}C which indicates that the sample processing window should be lower than 315 ^{o}C.

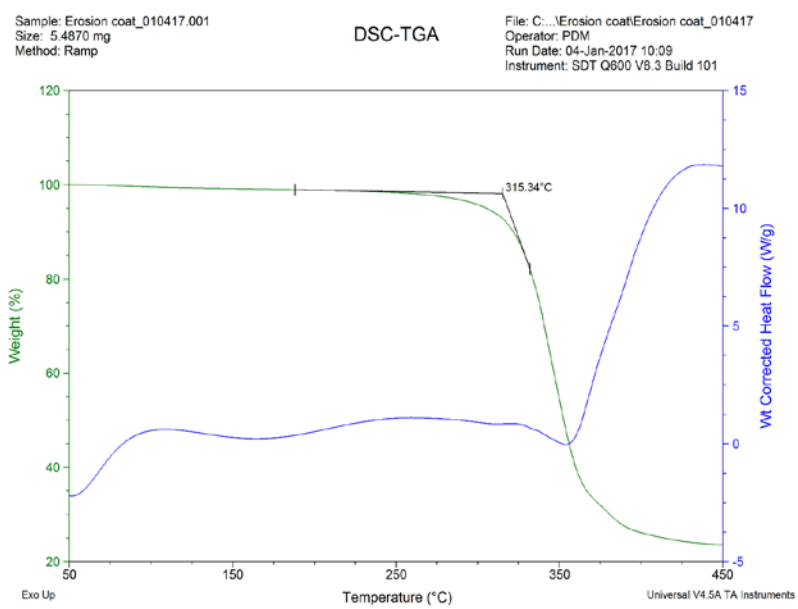

Fig. 9. TGA and DSC curves of rain erosion coating material

3.2 Task 2: Bond/Adhesive Strength Measurements of treated and untreated substrates

In this task, we characterized the bond strength of elastomeric coatings on to the base substrate, before and after IR, Sonication and simultaneous IR-Sonication treatments, with adhesion-in-peel test by peeling the surface coat from the substrate at a specified rate. During the IR application, the temperature at the bondline was monitored with the help of embedded thermocouples coupled with LabVIEW. Infrared thermography of the backside of the test coupons during exposure to the infrared heating was used to fully map the temperatures experienced by the substrate. All the experiments were performed in triplicate.

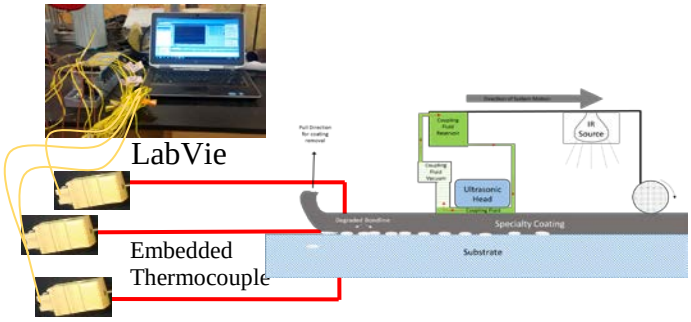

Fig. 10. Schematic of thermal profiling of the underlying substrate by employing embedded thermocouples.

We used embedded thermocouples, as shown in Fig. 10 and 11, to characterize the underlying substrate and measure the worst-case temperature experienced by the bond surface of the substrate. During this procedure the temperature at the top, bondline and bottom of the substrate was monitored with the help of LabVIEW. We have experimentally investigated the cross-sectional temperature profiles of the coat and the substrate at different IR intensities. Q-type thermocouples with shielded tip were attached at the bottom, interface and on the top the coated substrates.

The Maximum IR intensity of the emitter unit used for this experiment was 4800W. The approximate area of incident was 8” X 8” surface. IR radiation with varying intensities, 50%, 75% and 100%, was applied for a period of 20 seconds and the temperature profile across the substrate (AFRL-RE 0.5 mm) was monitored using LabView. Figures 12-14 show temperature profile across the substrate during IR radiation exposure of varying intensities. At 50% IR intensity and after 18 s exposure, the temperature at the top, interface and bottom of the substrate is temperature is 124 ^{o}C, 84 ^{o}C and 50 ^{o}C, respectively. This demonstrates that at this intensity the substrate could be exposed for 18s without adversely affecting the top and the underlying substrate. Similarly, exposure time for different substrates were determined at 75% and 100% IR intensities.

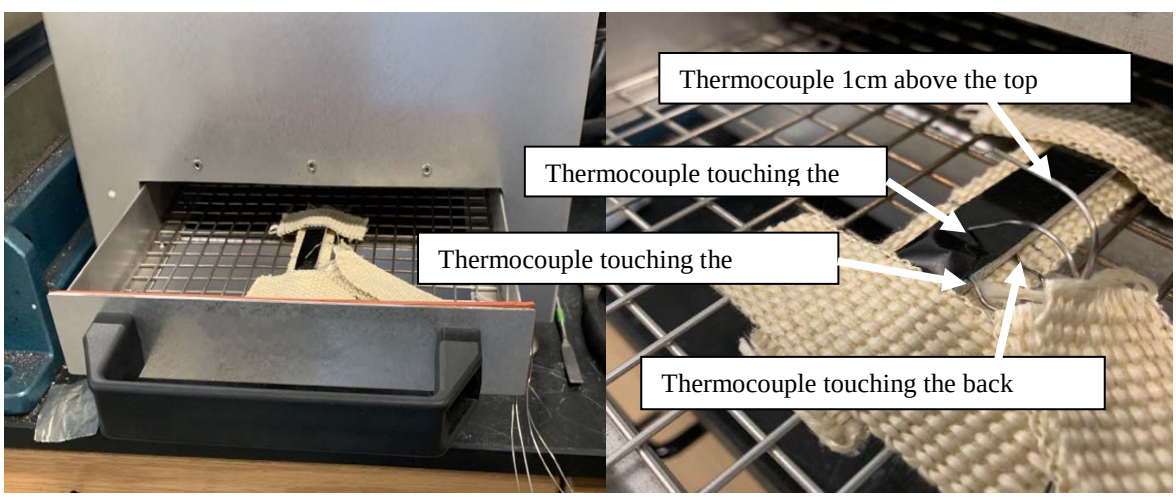

Fig. 11. Thermocouple arrangement for temperature profile monitoring

As evident from the Fig. 12-14, the temperature difference between top surface and interface were in the range of 45-55°C. Interface temperature were in the range of 90°C-110°C. The point where temperature difference between the topcoat and the interface was maximum was labeled as del T (ΔT) and chosen as the optimum exposure time need to induce highest shear stress. The time required to achieve the maximum ΔT was found to be 18s, 12s and 8s for 50% intensity, 75% intensity and 100% IR intensity, respectively. Similarly, we have also determined the exposure time for other substrates; 1 mm RE and 0.5 mm and 1 mm FP coatings.

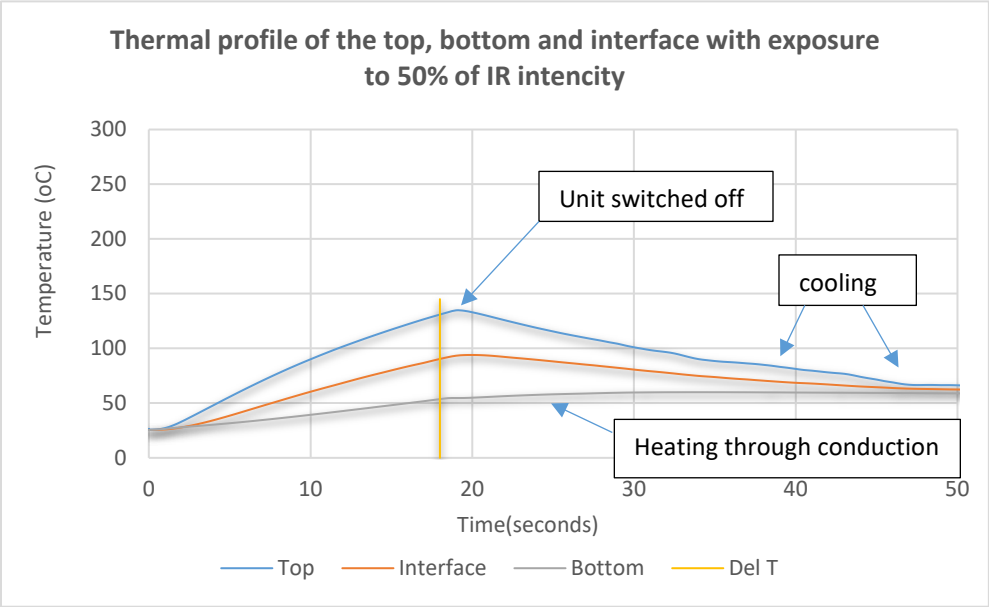

Fig. 12 .Temperature profile across the substrate during 50% IR intensity exposure for a period of 20s

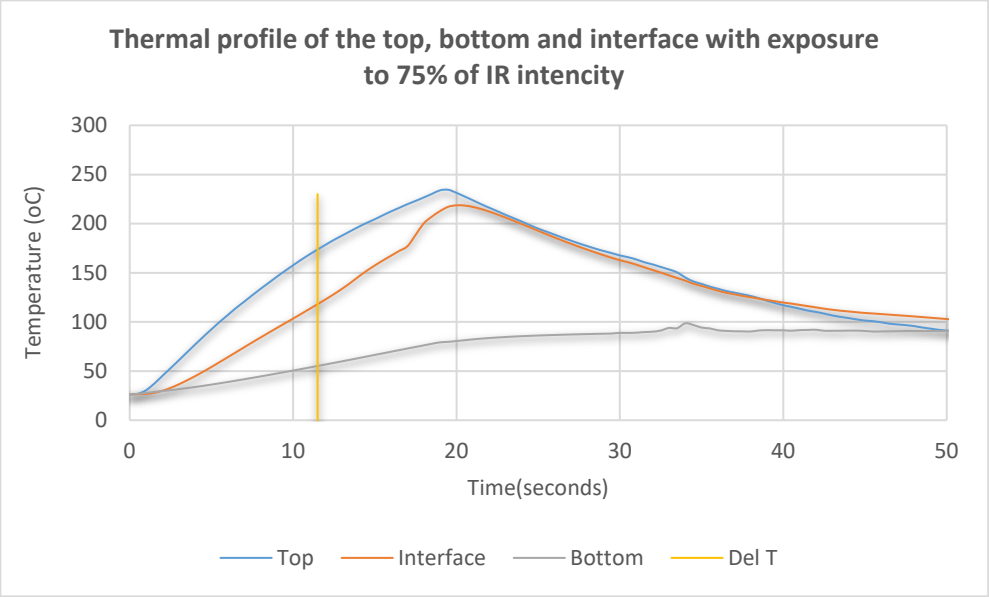

Fig. 13. Temperature profile across the substrate during 75% IR intensity exposure for a period of 20s.

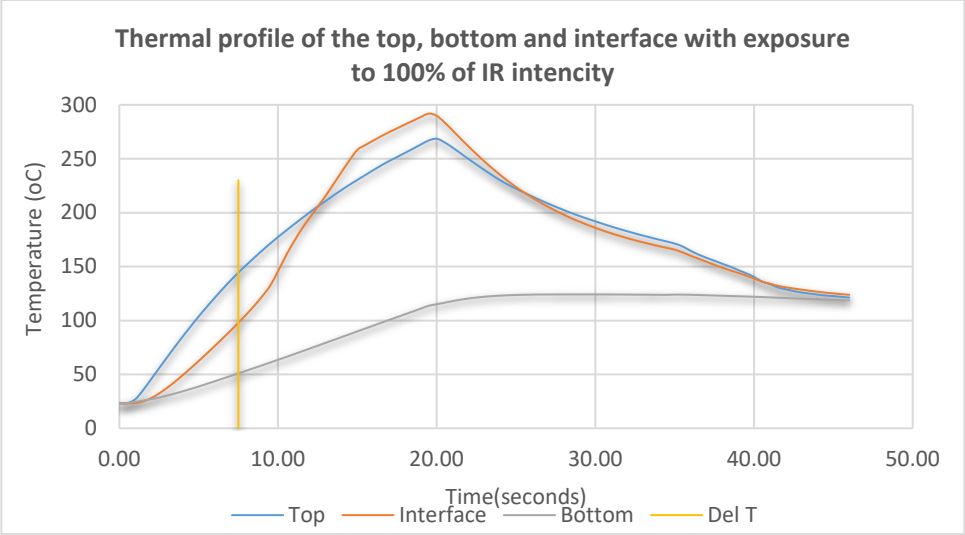

Fig. 14. Temperature profile across the substrate during 100% IR intensity exposure for a period of 20s.

3.2.1 Peel Test for determining the reduction in interfacial bond

Adhesion strength (defined as the maximum force divided by the width of the specimen) measurement before and after IR was determined according to the ASTM D429 standard (Standard for Rubber Property-Adhesion to Rigid Substrates). In this standard peel-test, bond strength between the elastomeric coating and the underlying rigid substrate was measured for the as-received and treated specimens. To initiate a peel-test, the samples before and after testing was inserted into the fixture of the peel test instrument. The free-end (The elastomeric coating was peeled with the help of a vise grip to provide a free-end at one end of the specimen) of the coating was inserted into an appropriate wedge action grip. The grip, connected to the crosshead, was driven in the tensile direction and the coating was peeled from the underlying substrate. The force required to peel the coating was recorded which gave a direct measurement of bond strength between the coating and the substrate (Fig. 15).

Fig. 15. Peel strength tester connected with LabView

3.2.2 Medium wave (MW) and shortwave (SW) IR exposure

Based on the temperature profile experiments, MW-IR exposure time for various samples was kept between 10 to 12s. Samples exposed for 10-12s showed surface temperatures between 85°C and 140 °C. These were shown higher reduction than other higher exposed samples. For the SW-IR, exposure time was kept between 5-8s. The recorded surface temperatures were in the range of 90 °C to 130°C. Approximate heat flux generated by SW heaters were around 9.7kW/ft^2 and in MW heaters were around 5.2kW/ft^2. Incident IR energy on the surface of samples due to SW-IR were between 0.51-0.71kJ, whereas due to MW-IR, were around 0.55-0.65kJ.

AFRL (0.5mm coating thickness on an Al substrate), NAVAIR and in-house samples of 3”x 0.5” dimensions were cut and exposed to SW-IR and MW-IR for the above-mentioned duration. Immediately after exposure, the sample was loaded on the peel-tester and peel strength was recorded, Fig. 16-17. It was found that average peel reduction achieved with SW-IR was around 76% or 3.6 times lower than the untreated samples. In the case of MW-IR exposure, the average peel reduction was 4 times lower than the as-received samples. It is pertinent to mention here that the slant increased force observed after the deadhesion point (Fig. 16) is due to the cooling of the substrate (heat loss to the surrounding) while it is being peeled in the machine.

We also tested the effect of the SW-IR and MW-IR on the in-house coated samples and found that the average reduction in peel was about 3-4 times lower than the untreated samples. The sealant samples from NAVAIR were of irregular thickness and had a lower as-received peel strength than AFRL coated samples. In this case, the peel strength reduction was found to be in the range of 50% with SW-IR exposure.

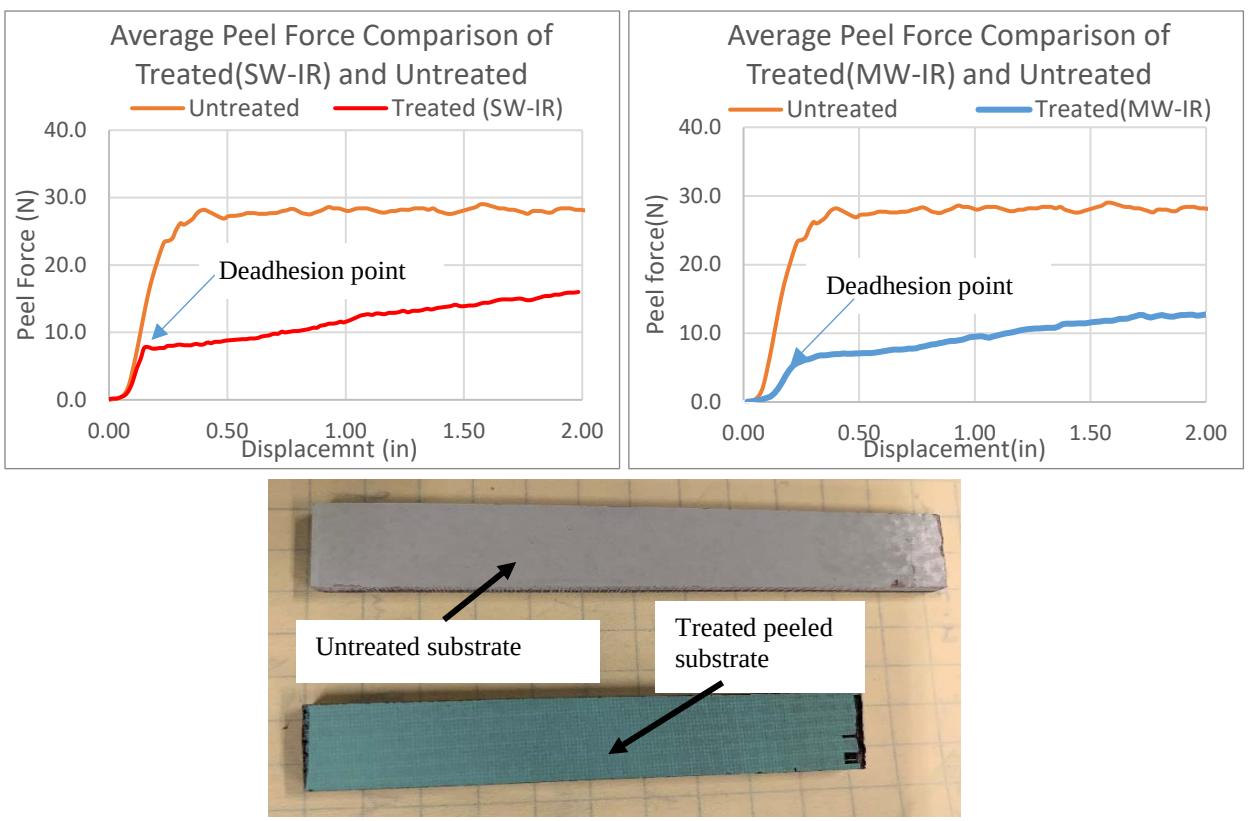

Fig. 16. Average peel for the AFRL-RE composite substrate, Coating thickness, 0.5 mm, before and after exposure

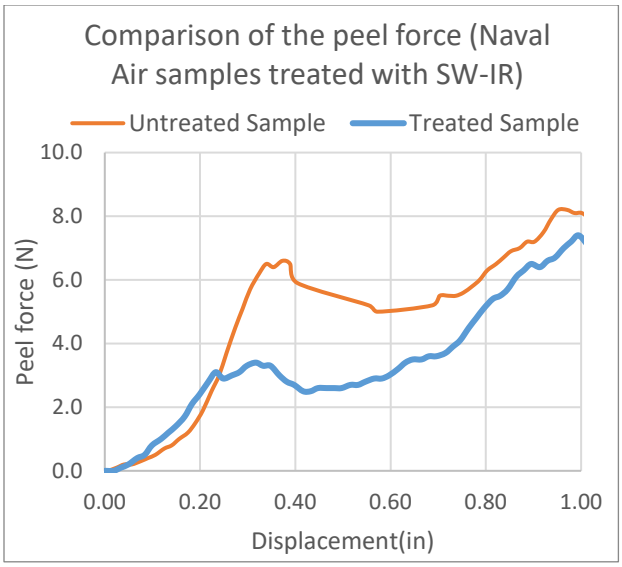

Fig. 17. Average peel for the NAVAIR sealant samples before and after SW-IR exposure

Next, we tested AFRL samples of 1mm thickness (3”x 0.5” dimension). Both, the RE and FP coated aluminum substrates were exposed to SW-IR for 12s. As-received 1mm Al-RE samples have twice the bonding strength (62N compared to 28N) than 0.5 mm thick composite substrate. Interestingly, the peel strength reduction after SW-IR exposure is more than 3 times when compared to the as-received samples (Fig. 18).

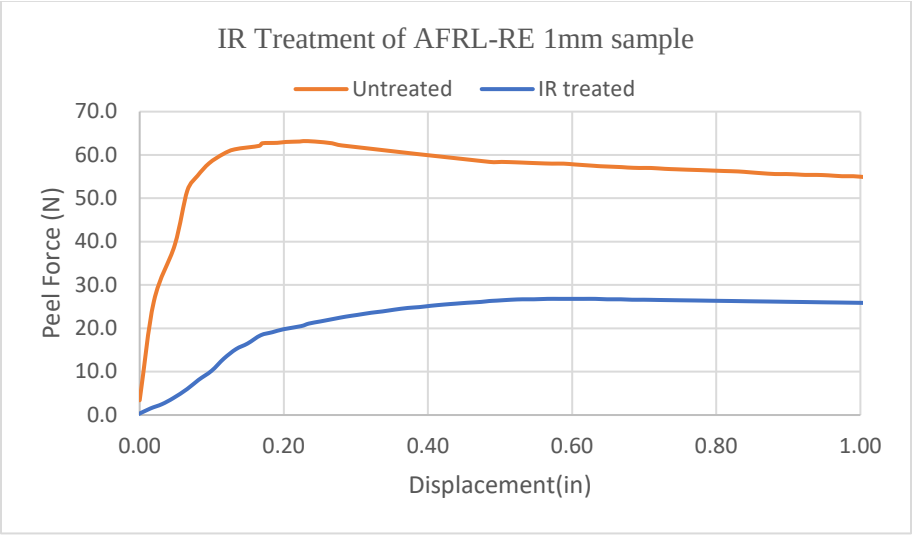

Fig. 18. Average peel for the AFRL-RE Al substrate, Coating thickness, 1 mm, before and after exposure

We also tested the peel of as-received AFRL-FP coated samples, 1mm thickness, and found that it had a higher bonding strength between the elastomeric coating and the underlysing substrate than compared to the RE coatings of the the same thickness. On exposing these samples to SW-IR for 10s and 12s, the peel strength reduction was found to decrease by more than 3.5 times (Fig.

19). 1mm thick coated top layer needed more exposer time (12S), and absorbed higher thermal energy, compared to the 0.5 mm thick coated top layer (8S). Compared to the sample with 0.5 mm thick topcoat, heat loss from the sample with 1 mm thick topcoat was slower. Therefore, cooling was apparently slower in these samples which resulted in a consistent peel strength after the deadhesion point. In the actual process, treatment and peeling will be performed simultaneously so no increase in the peel strength is expected after the deadhesion point. This was the case when the samples were peeled with a hand-held equipment rather than placing and peeling in the peel tester machine.

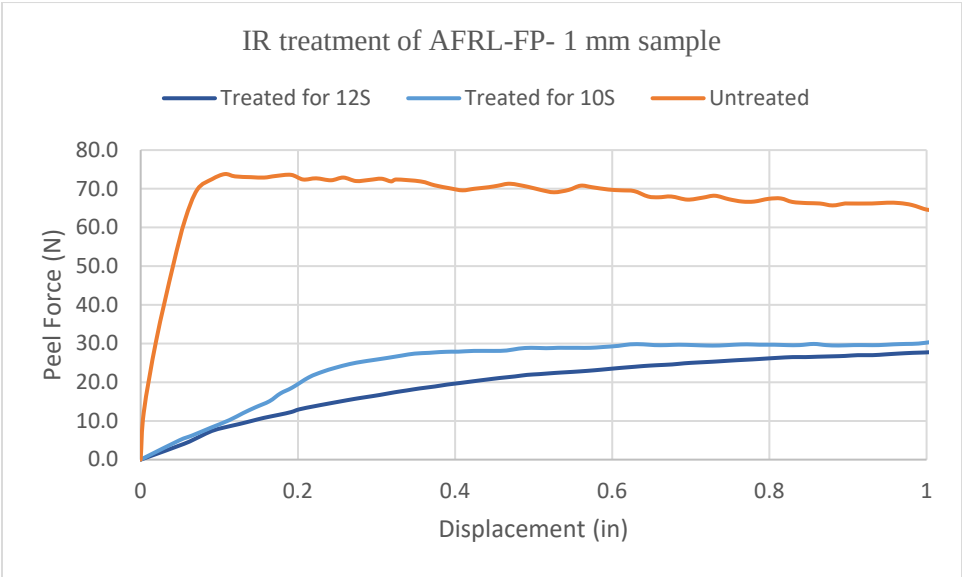

Fig. 19. Average peel for the AFRL-FP Al substrate, Coating thickness, 1 mm, before and after exposure

We received another set of RE coated composite substrate with different filler additives. We faced many challenges while working with these composite substrate samples. Due to the abrasiveness of the composite material, it was very difficult to cut and machine these samples. After a lot of effort, we managed to get a few samples of 0.5 mm thickness RE coated substrate. On exposure to SW-IR, the reduction in the peel strength was greater than 3 times, i.e., in the range of 60-75%. Both IR exposure of 8 S and 10 S gave similar results. **It is pertinent to mention that we have successfully achieved our target of 50% reduction in the bonding strength by exposing the substrates to IR radiation only.**

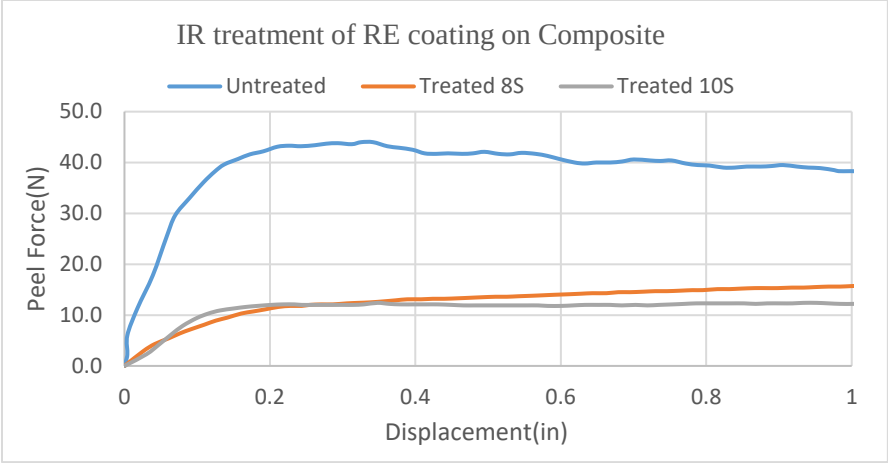

Fig. 20. Average peel for the AFRL-RE composite substrate, Coating thickness, 0.5 mm, before and after exposure

Encouraged by the success of the experiment, we tried removing the 1 mm thick AFRL-FP substrate of 6”x6” dimension (Fig. 21.) The sample was exposed to SW-IR for 15-18s and the topcoat was manually peeled with the help of pliers. The topcoat was easily removed after exposure, which indicate that IR exposure alone is sufficient to remove these thick elastomeric coatings from aluminum and composite underlying substrates.

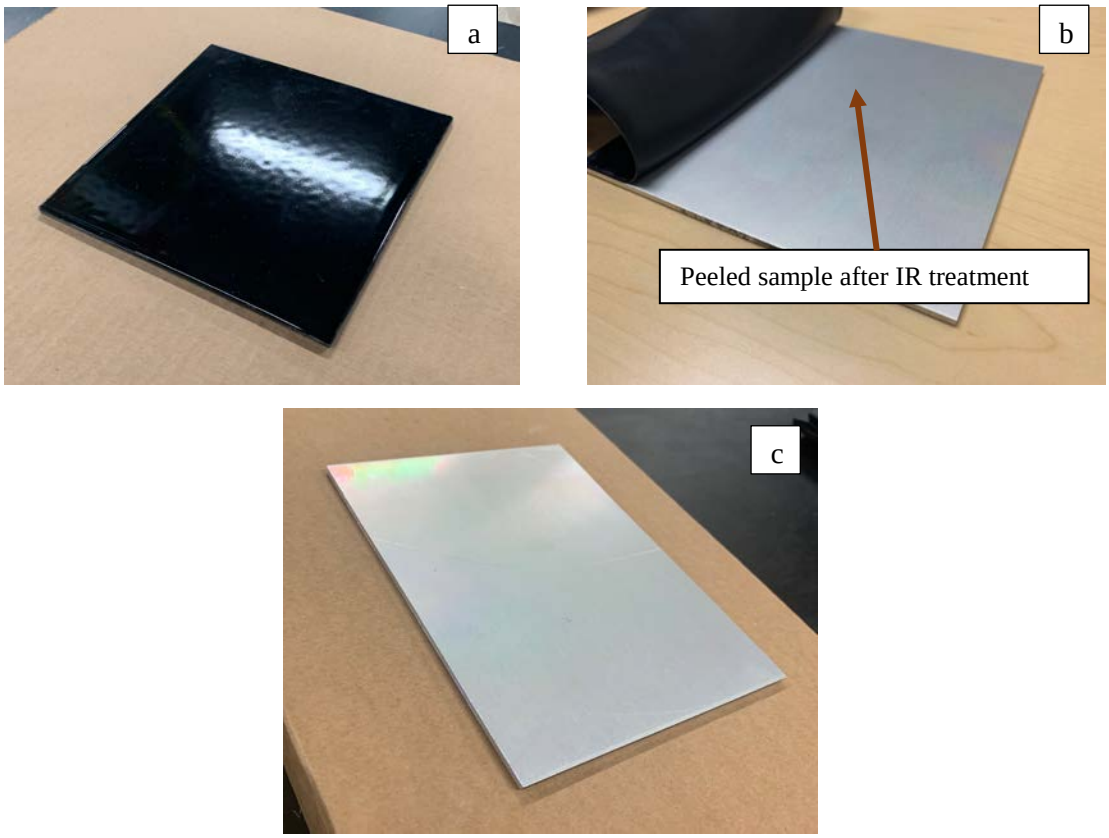

Fig. 21. 6”x6” AFRL-FP sample before(a) and after peel (b,c)

3.2.3 Computational Simulation

We also created a Transient model in ANSYS to analyze the IR treatment process. The Analysis was done using two separate models. First model takes the IR heating rate as the input to calculate the temperature data for the transient model. Second model takes the transient data to generate the thermally induced stresses (Fig. 22). Transient temperature data was generated for a 10s period. During this time the heating rate was constant. Heating was uniform for the selected surface strip during that period. The model predicted that the maximum shear stress was seen in the interfacial layer (Fig. 23) which is in line with our experimental results.

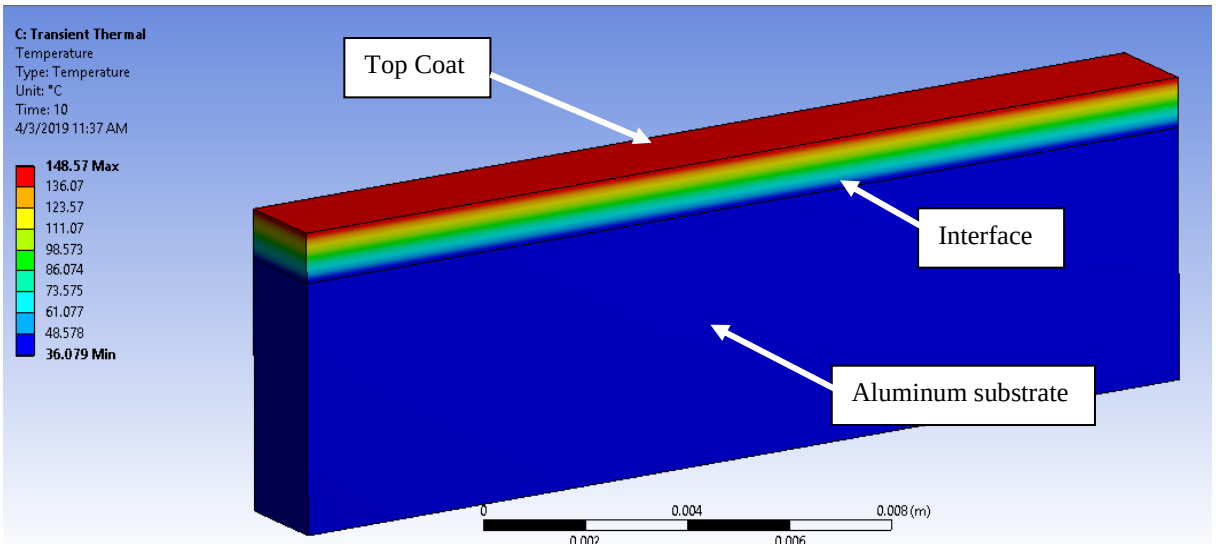

Fig. 22.Transient temperature variation across the cross section

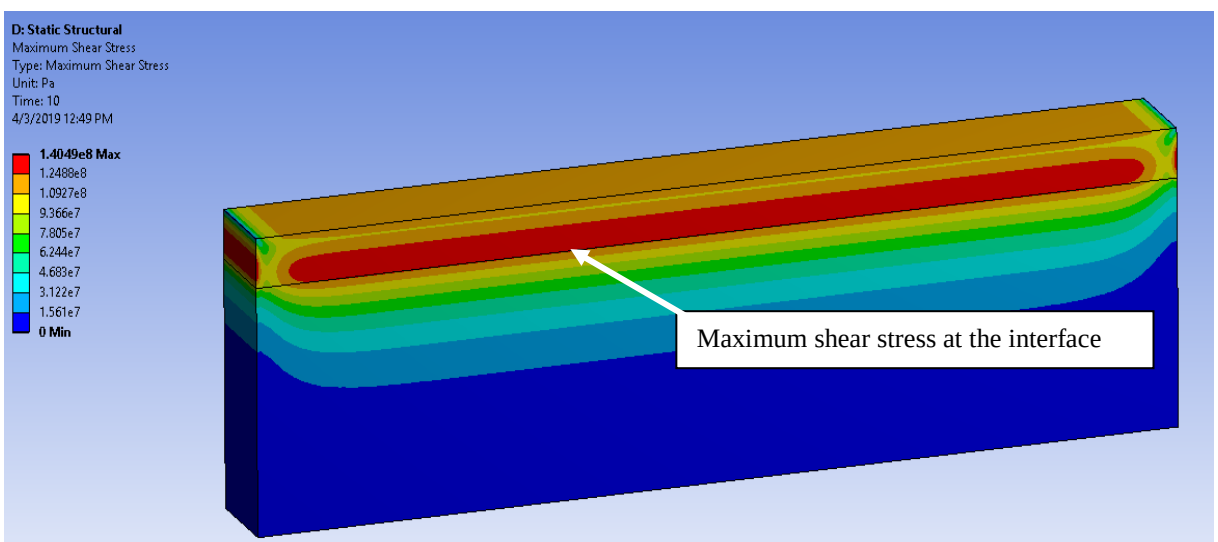

Fig. 23. Shear stress distribution across the cross section.

3.2.4 Infrared Thermography

One of the major concerns for any coating removal technology is to minimize damage to the underlying substrate material. Apart from the experiments using thermocouples to acquire cross-sectional temperature data, we have also performed infrared thermography of the front and backside of the test coupons right after the exposing to the infrared heating. FLIR E5 model thermal imaging camera (Fig. 24) was used to take the images. The images were taken with in a two second window from taking the sample out from IR heating. There were some challenges in using this equipment for this application as higher temperature ranges tend to create blurry surfaces and edges due to the continues heat loss happening from the surfaces. We used IR exposure for 5s with maximum intensity to monitor the temperature at the top and bottom of the surface with IR thermography.

Fig. 24. FLIR E5 Thermal Imaging camera

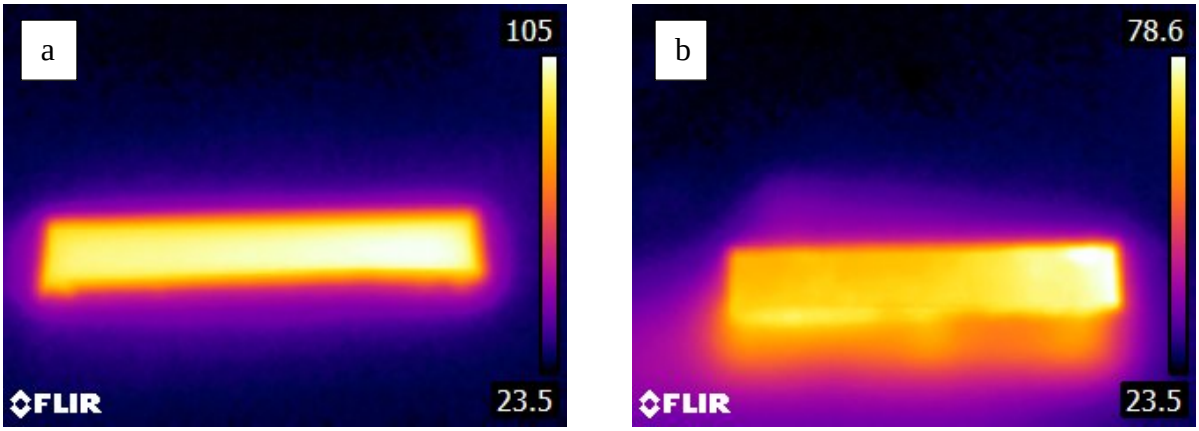

Fig. 25. Thermal imaging after heating for 5S with full IR intensity a) top surface b) bottom surface

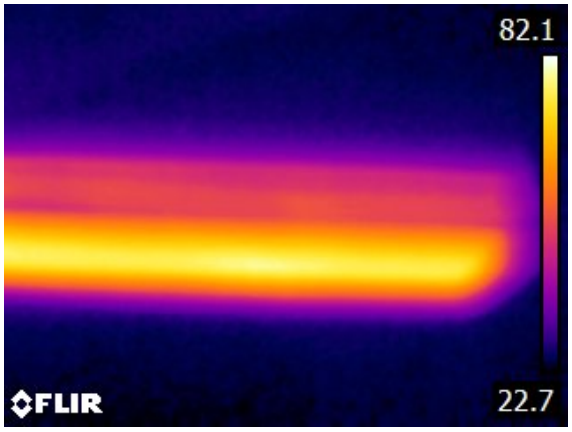

Fig. 26. Thermal imaging of the cross section after heating for 5S with full IR intensity

Top surface was showing temperatures in the range of 105ºC whereas the bottom surface was showing temperature in the range of 78ºC. Cross sectional images were too blurry as a result of the surrounding convectional heat loss. In general, there was a clear gap in temperature on the top coat, and the bottom substrate surface.

3.2.5 Ultrasonication

We investigated the use of ultrasonication in the coating removal application. An ultrasonic (welding) machine (XPE-1200-110) of 20kHz in frequency and 1.2kW in power was purchased and used for experimentation. (Fig. 27).

Fig. 27. Ultrasonic machine (XPE-1200-11)

Two types of experiments were carried out.

1) Directly applying ultrasonic energy to the top layer of the sample.
2) Maintaining a thin film of water layer in between the ultrasonic horn and the topcoat.

The first approach was to use friction stir heating and separation due to itself. In this method, the ultrasonic horn makes a solid contact with the top layer and transfer sonication energy through the

top layer and create stress at the interface of topcoat and the underlying substrate. The second approach was to induce cavitation and remove the top coat by putting a layer of water in between the ultrasonic horn and sample. The gap between the top surface and ultrasonic horn is small and in the range of 0.1 - 0.5mm.

Fig. 28. Ultrasonication for removing sealants.

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Both methods were able to remove a significant amount of top layer coatings. Ultrasonication was most successful in removing thick elastomeric sealants from the NAVAIR samples. Fig. 28 and 29 demonstrate the experimental setup for applying ultrasonication to a sealant samples. Ultrasonication (20kHz , 1200W) was applied to the sample to remove the top coat while dispensing a continuous stream of water over it. Water was used to create a jet of high energy water droplets that introduce cavitation wear at the interface. The use of water also prevents overheating of the sample. This technique is useful for removing sealants from fuel tanks as a hand-held ultrasonication device can be easily operated by a technician from the inside of a fuel tank.

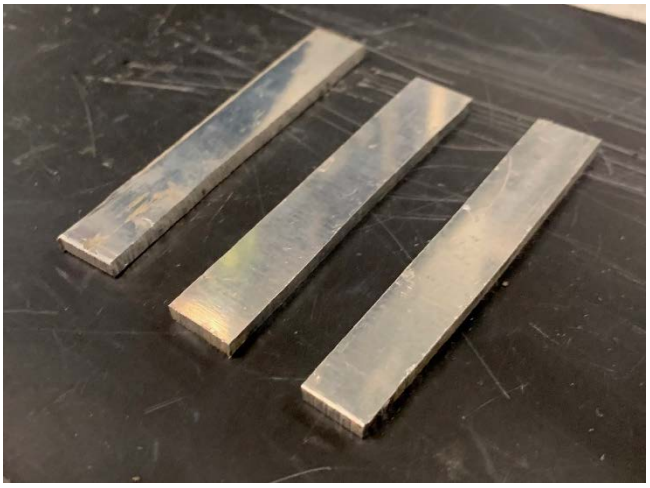


Fig. 29. Top coat removed samples

3.3 Task 3 Fabrication and Testing of IR-Sonication probe

3.3.1 IR-Sonication synergy: Hybrid approach

We next tried the synergic effect of simultaneous application of Sonication and IR. The AFRL-RE samples were first subjected to ultrasonication and then SW-IR exposure and it was found that the reduction in the peel strength was more than the IR and sonication alone (Fig. 30). The synergic effect of IR-Sonication approach has been presented in the Table 1.

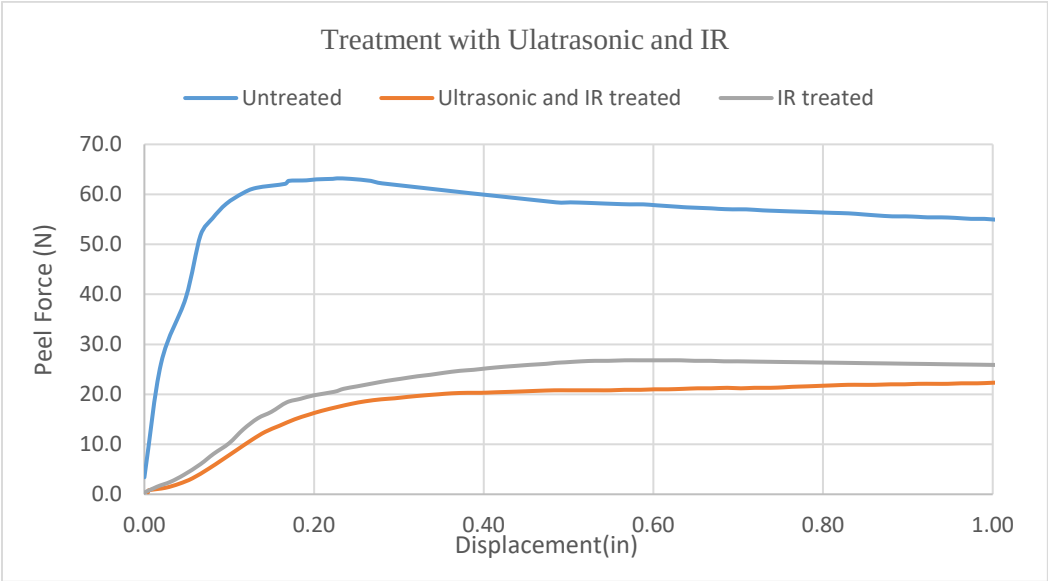


Fig. 30. IR-sonication exposure effect on peel strength of AFRL-RE sample of 1 mm thickness

Table 1. Comparison with different treatments

Thickness	Untreated (N)	IR treated (N)	Ultrasonic Treated (N)	Hybrid (N)
AFRL-RE -20 mil	35-45	12-15	30 -35	11-13
AFRL -RE-40 mil	60-65	20-22	45-40	18-20

Additional percentage reduction of 5-10% was observed with the hybrid approach compared to the best-case IR alone approach. We have tried several scenarios with different IR and sonication sequence. Introducing ultrasonication first and then IR gave was operationally easier than exposing to IR first and then ultrasonication.

3.3.2 3D model of the sonic-IR hybrid lab-scale prototype.

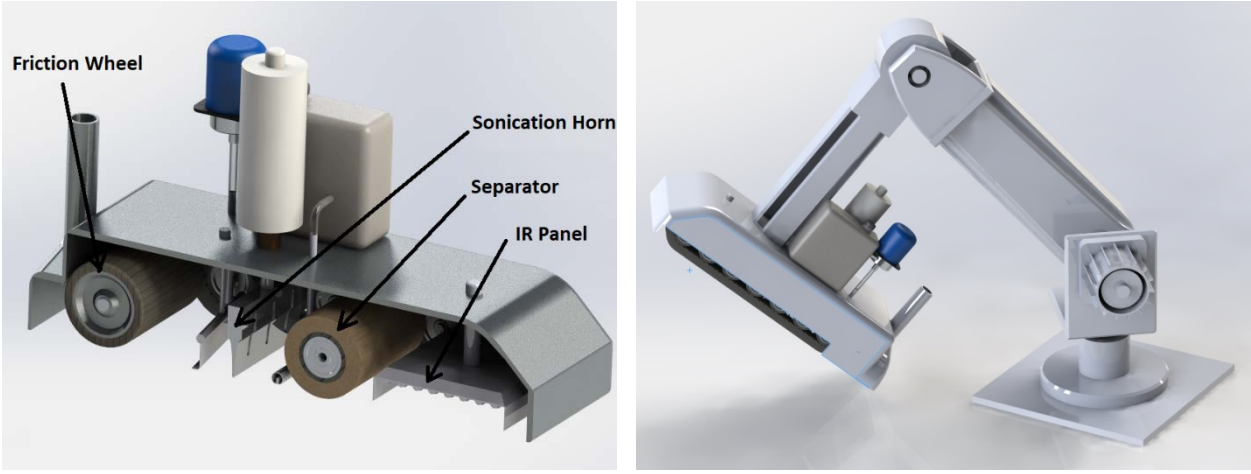


Fig. 31. 3D model of the sonic-IR hybrid lab-scale prototype.

A portable unit as well as a unit operated by a robotic arm were modelled in SolidWorks (Fig. 31). This unit capable of providing IR or ultrasonic independently or simultaneously. IR panel can be made in an easily interchangeable manner to switch between SW-IR heater cassette and MW-IR heater cassette depending on the requirement.

3.4 Task 4.1 Characterization of Surface coat after treatment.

3.4.1 Structural integrity

One of the key goals of this project was to ensure that the coating removal approach adopted here has no detrimental effect on the underlining substrate. In order to determine any adverse effect on the underlying substrates, we performed thermogravimetric analysis (TGA), FTIR and SEM imaging of the treated/peeled (Fig. 32) and as-received samples. were carried out to investigate the after effects.

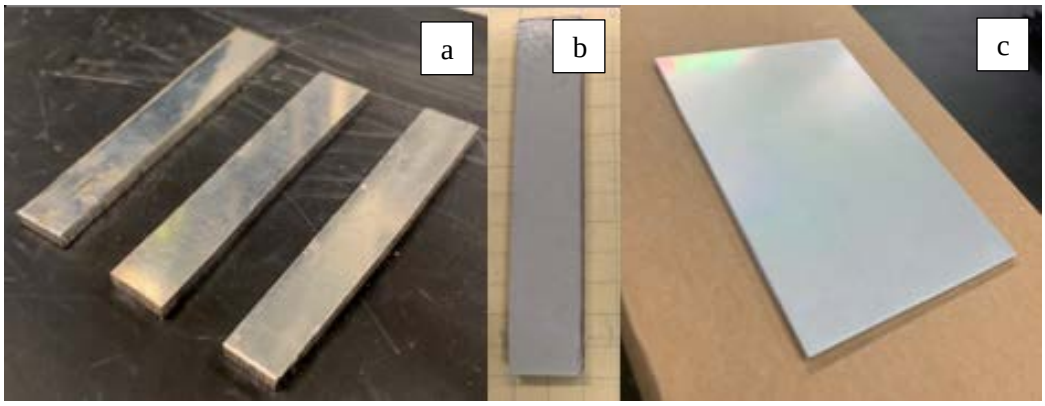


Fig. 32. Peeled substrates a) NAVAIR samples b) Composite sample c) 6”X6” Aluminum sample

3.4.2 FTIR Analysis

FTIR analysis was performed on the treated and the untreated samples. As evident from the spectra (Fig. 31) the treated and untreated underlying composite substrates have the same absorption peaks throughout the spectral range, which clearly indicates that there is no change in the chemical makeup of the material. It is indicative of the fact that the IR exposure of samples had no adverse effect on substrate.

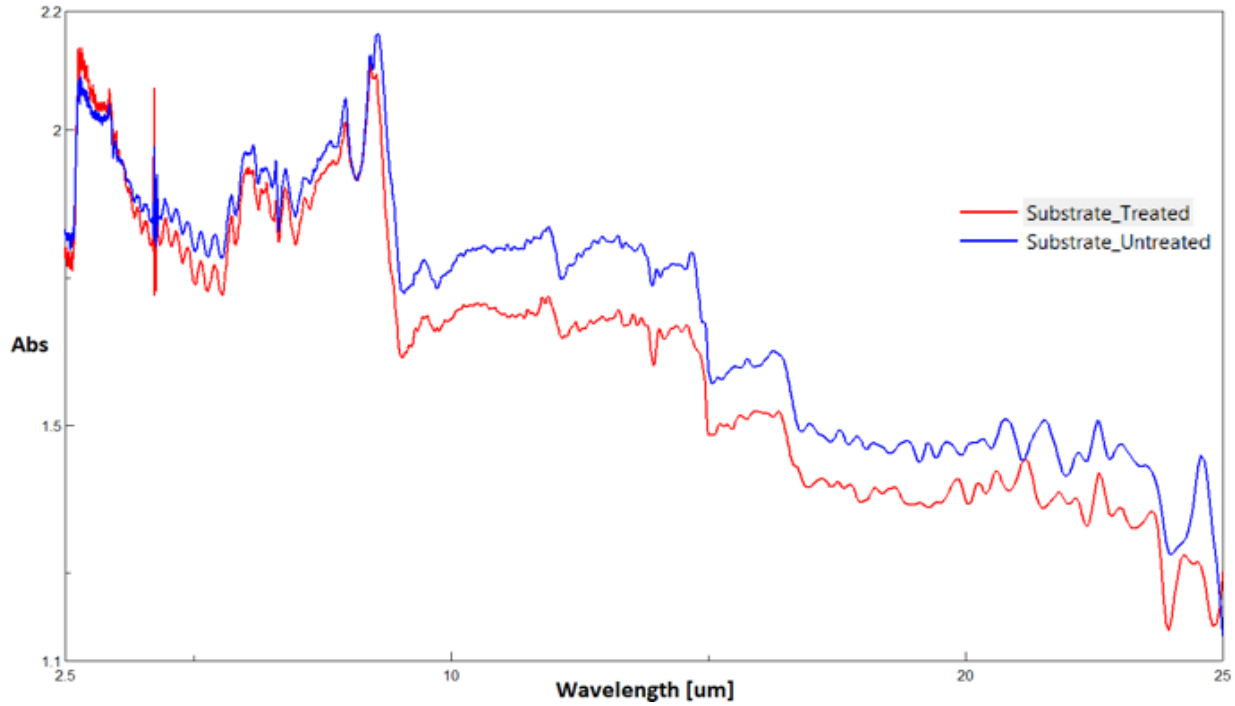


Fig. 33. FTIR spectra of treated and untreated composite substrates.

3.4.3 TGA Comparison

Thermal analysis studies of specialty elastomeric coating and underlying substrates (epoxy composite and bismaleimide composite) was performed. According to the TGA results, IR treated and untreated samples have shown no difference at all. Not only the substrate, even the top coat, where it was directly exposed to IR, has shown no difference at all. This is because the operating temperatures of these samples which were exposed to IR were significantly lower than their decomposing temperatures.

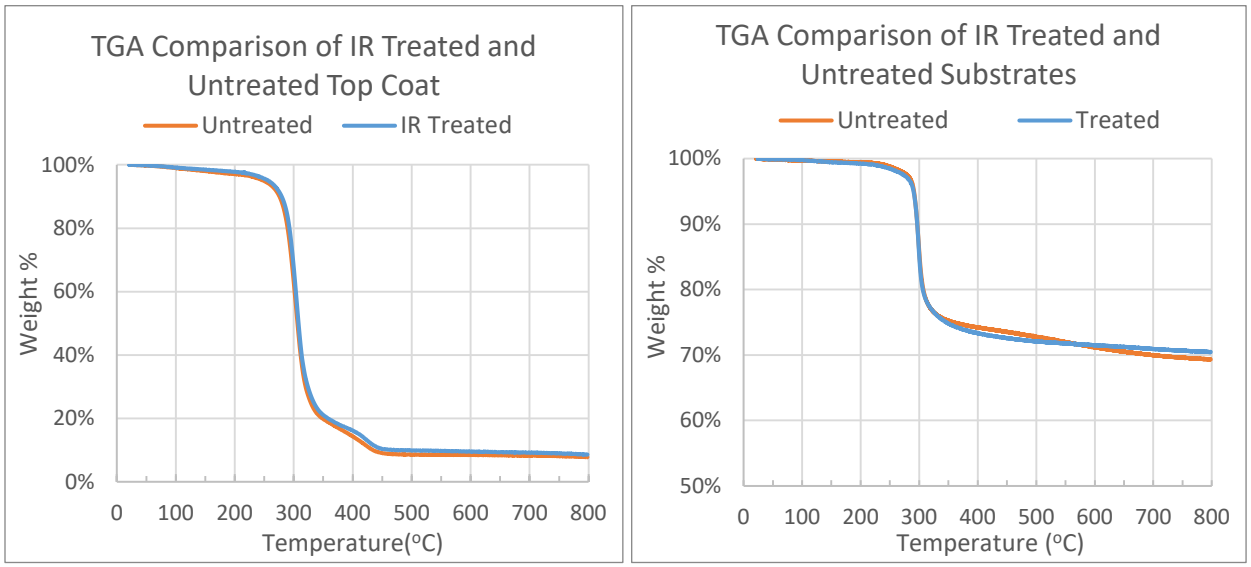
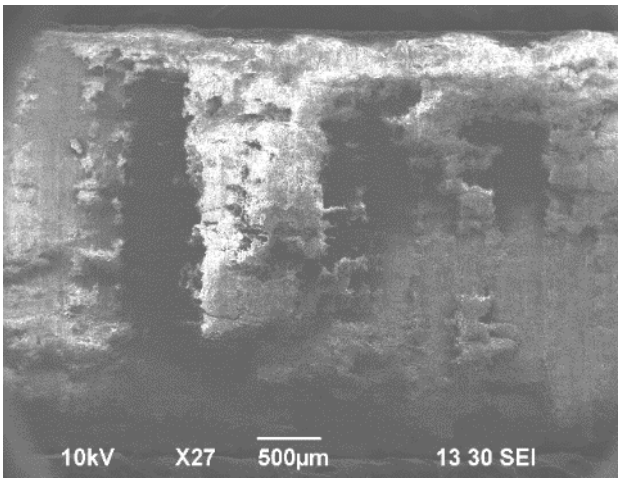


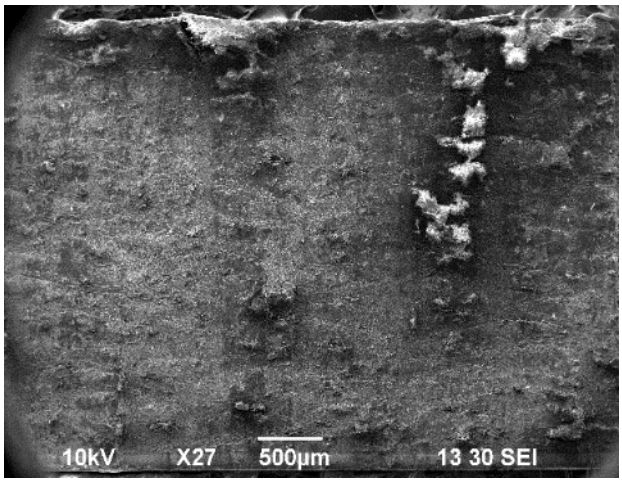
Fig. 34. TGA of treated and untreated topcoat and underlying substrate

3.4.4 SEM Image Analysis

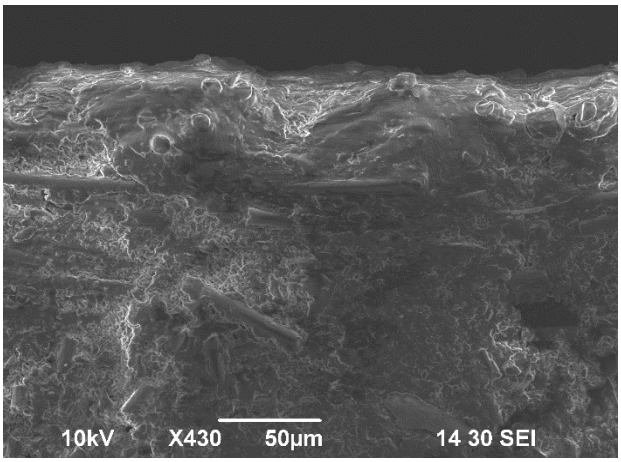
SEM imaging of the treated and as-received AFRL composite substrates was done to determine if any crack or delamination was introduced in the sample due IR exposure. As per the SEM images, we didn't see any delamination or any kinds of cracks inside the substrate after treating with IR. This clearly establishes that our process has no adverse effect on the underlying substrate.



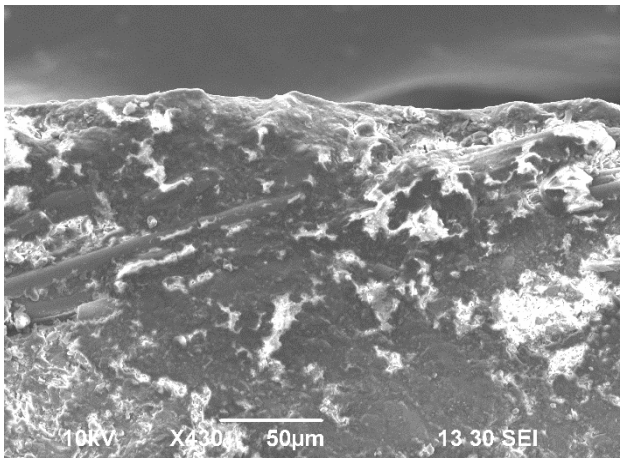
Cross section of untreated substrate



Cross section of IR treated substrate



Top surface of untreated substrate



Top surface of IR treated substrate

Fig. 35. SEM images of treated and untreated underlying composite substrates.

3.5 Uncertainty Analysis

To check the repeatability of the results, at least 3 trials were carried out with same experimental conditions. Observed data showed minimal deviation. IMADA Digital Force gauge which we used for ASTM D429 Method B has an accuracy of +/- 0.2 %. Considering the range, the measurement uncertainty for the recorded force was 0.16 N. K type thermocouple uncertainty was 1.5^{o}C for the operation temperature range.

3.6 Cost Analysis

3.6.1 Basic Operational Cost

Considering the experiments carried out in the lab basic operation cost was calculated. This was in regard to the average energy consumption. To remove one square feet of coating using IR treatments, the energy required was in the range of 100-200 kJ/ft^2. This is accounted to $0.001-0.004 /ft^2 for an electricity tariff of $0.065/kWh [13]. Table 2 shows the energy cost per square feet of removal using IR treatments. Using ultrasonic(1200W) gives energy consumption of 200-300kJ/ft^2 and would account to $0.002-0.006/ft^2.

Table 2. Operation cost of IR heating

IR Energy (W)	Effective surface heating area (in^2)	Average Time of Exposure (S)	kJ/ft^2	Tariff ($/kWh)	Cost ($/ft^2)
4800	64	8	86.40	0.065	0.0016
4800	64	10	108.00	0.065	0.0020
4800	64	12	129.60	0.065	0.0023
4800	64	14	151.20	0.065	0.0027
4800	64	16	172.80	0.065	0.0031
4800	64	18	194.40	0.065	0.0035
4800	64	20	216.00	0.065	0.0039

4 CONCLUSION

We have successfully developed a non-chemical and non-media radiation-based technology that can remove thick elastomeric coatings from the aluminum and composite substrates without damaging the underlying substrates while being ergonomically less challenging. All deliverables were delivered in a timely manner.

- **Final Deliverable 1**: Developed and tested a tailored Infrared-Sonication hybrid technology for introducing interfacial bond failure between the elastomeric coating and the underlying substrates.
- **Final Deliverable 2.** Achieved more than 50% reduction in the peel-strength of the elastomeric coatings after IR alone and IR-Sonication treatment.
- **Final Deliverable 3**: Performance and cost analysis of IR-Sonication hybrid technology by testing, and characterization of substrate materials after IR and IR-Sonication treatment was done.

The IR and sonication treatments were carried out for several different specialty coatings from AFRL and NAVAIR. The key significant deliverable of introducing interfacial bond failure

between the topcoat and the underlying substrate was accomplished (**Milestone 2.1 and Final Deliverable 2**) with a far better results than that was proposed in the proposal (25-50% bond strength reduction). On an average, 65-75% peel-strength reduction was achieved by the use of tailored IR treatment alone and IR-Ultrasonic combination. Both, the shortwave infrared (SW-IR) and mediumwave infrared (MW-IR) treatments demonstrated peel-strength reductions in the range of 75-50% with different exposure time. The desired peel strength reduction was achieved significantly quicker with SW-IR than that achieved with MW-IR exposure. Optimum SW-IR exposure for a 05" x 3" sample of 0.5 mm thickness was 8 seconds whereas for a same size sample of 1.0 mm was 12 seconds.

In order to determine any adverse effect on the underlying substrates, thermogravimetric analysis (TGA), FTIR and SEM imaging of the treated/peeled and as-received samples were investigated. AFRL-RE aluminum and composite substrates of 0.5 mm and 1 mm thick coating, AFRL - FP 1mm thick coating, NAVAIR samples were some of the samples tested. The results clearly established that the developed technology has no detrimental effect on the underlying substrates.

The developed coating removal technology is an environmental-friendly method as it does not produce any toxic gas or suspended solid waste in the air. Operation cost for this technology is very low compared to the conventional technologies currently being used. Also, interfacial bond reduction is achieved at a much lower temperature when compared with energy-intensive technologies such as laser ablation. In conclusion, we have successfully addressed SERDP's goal to develop a large-scale non-chemical, non-media removal process for thick, elastomeric coatings and treatments, used on Department of Defense Weapon Systems.

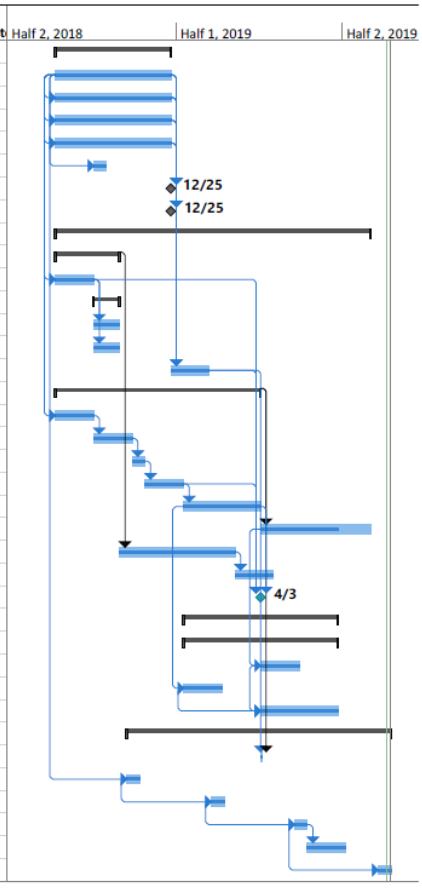
4.1 Future Work

We will develop a fully automated system with thickness sensor feedback that is capable of removing coatings on a larger scale (over 3 feet panel length). Design and fabricate a miniature unit having a single IR lamp, instead of a lamp caste, to remove coating from hard to reach areas on an aircraft such as corners and sharp curvatures. Design and fabricate hand-held portable ultrasonic device to remove sealants from the fuel tank.

Project planning

Project plan was created using Microsoft Project with all the tasks, subtasks and milestones. The task progress tracking and monitoring was carried out daily.
A thirty-minute monitoring and planning meeting was held between the PI and the main postdoc at 10.00am on every working day, to plan the day's work and to discuss the progress of the previous day's work. Weekly one-hour meeting was held on every Tuesdays (9.30-10.30am) to discuss the weekly progress in detail. Meetings with AFRL personnel were held from time-to-time to discuss sample preparation work streams. As a result of this rigorous monitoring, all the tasks were accomplished in a planned timely manner.

ID	Task Name	Duration	Start	Finish	% Complete
1	**1 Characterization of the surface coat and the underlying substrate**	**90 days**	**Wed 8/22/18**	**Tue 12/25/18**	**100%**
2	1.1 FTIR and Specular reflectance for polyurethane-filler composite	90 days	Wed 8/22/18	Tue 12/25/18	100%
3	1.2 FTIR and Specular reflectance for the underlying substrates	90 days	Wed 8/22/18	Tue 12/25/18	100%
4	1.3 DSC analysis of surface coat polyurethane-filler composite	90 days	Wed 8/22/18	Tue 12/25/18	100%
5	1.4 DSC analysis of underlying substrates	90 days	Wed 8/22/18	Tue 12/25/18	100%
6	1.5 XPS Analysis of the Coating	10 days	Wed 10/3/18	Tue 10/16/18	100%
7	1.6 M 1.1 Selectionof the optimum IR range	0 days	Tue 12/25/18	Tue 12/25/18	100%
8	1.7 M 1.2 Selection of the sonication range	0 days	Tue 12/25/18	Tue 12/25/18	100%
9	**2 Bond Strength Measurements of treated and untreated substrates**	**246 days**	**Wed 8/22/18**	**Wed 7/31/19**	**94%**
10	**2.1 Testing bond strength of samples before treatment**	**50 days**	**Wed 8/22/18**	**Tue 10/30/18**	**100%**
11	2.1.1 Identifying Testing Standards for Coatings/Adhesives	30 days	Wed 8/22/18	Tue 10/2/18	100%
12	**2.1.2 Developing In-house testing setup**	**20 days**	**Wed 10/3/18**	**Tue 10/30/18**	**100%**
13	2.1.2.1 In-house Pull Test Setup	20 days	Wed 10/3/18	Tue 10/30/18	100%
14	2.1.2.2 In-house preparation of polyurethane coated samples	20 days	Wed 10/3/18	Tue 10/30/18	100%
15	2.2 Testing bond strength of after IR treatment	30 days	Wed 12/26/18	Tue 2/5/19	100%
16	**2.3 Testing bond strength after ultrasonication**	**160 days**	**Wed 8/22/18**	**Tue 4/2/19**	**100%**
17	2.3.1 Establishment of industry partner for Ultrasonic testings	30 days	Wed 8/22/18	Tue 10/2/18	100%
18	2.3.2 Sending samples to carryout ultrasound tests	30 days	Wed 10/3/18	Tue 11/13/18	100%
19	2.3.3 Getting quatations and finalizing a Ultrasonic machine	10 days	Wed 11/14/18	Tue 11/27/18	100%
20	2.3.4 Setting up the machine and getting the proper training	30 days	Wed 11/28/18	Tue 1/8/19	100%
21	2.3.5 Testing the samples(AFRL and In-house coated samples)	60 days	Wed 1/9/19	Tue 4/2/19	100%
22	2.4 Testing bond strength after IR-Sonication treatment	86 days	Wed 4/3/19	Wed 7/31/19	70%
23	2.5 Monitoring temperature profile across the underlying substrate	90 days	Wed 10/31/18	Tue 3/5/19	100%
24	2.6 Infrared thermography for spot temperature measurements	30 days	Wed 3/6/19	Tue 4/16/19	100%
25	2.7 M 2.1 50-25% reductionin the peel-strength	0 days	Wed 4/3/19	Wed 4/3/19	100%
26	**3 Fabrication and Testing of IR-Sonication system**	**120 days**	**Wed 1/9/19**	**Tue 6/25/19**	**100%**
27	**3.1 Design and Fabrication of IR-Sonication probe**	**120 days**	**Wed 1/9/19**	**Tue 6/25/19**	**100%**
28	3.1.1 3D Design of IR device	30 days	Wed 4/3/19	Tue 5/14/19	100%
29	3.1.2 3D Design of Sonication Device	30 days	Wed 1/9/19	Tue 2/19/19	100%
30	3.1.3 3D Design of the Integrated system	60 days	Wed 4/3/19	Tue 6/25/19	100%
31	**4 Final Design and Economic Analysis**	**206 days**	**Thu 11/8/18**	**Thu 8/22/19**	**100%**
32	4.1 Characterization of Substrate after treatment	1 day	Wed 4/3/19	Wed 4/3/19	100%
33	4.2 Quarterly Progress Report - 1	10 days	Thu 11/8/18	Wed 11/21/18	100%
34	4.3 Quarterly Progress Report - 2	10 days	Fri 2/8/19	Thu 2/21/19	100%
35	4.4 Quarterly Progress Report - 3	10 days	Fri 5/10/19	Thu 5/23/19	100%
36	4.5 Costing and Economic Analysis	30 days	Fri 5/24/19	Thu 7/4/19	100%
37	4.6 Final technical report	10 days	Fri 8/9/19	Thu 8/22/19	100%



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APPENDIX

Nomenclature

AFRL	Air Force Research Laboratory
NAVAIR	Naval Air System Command
DOD	Department of Defense
IR	Infrared Radiation
LW-IR	Longwave Infrared Radiation
MW-IR	Mediumwave Infrared Radiation
SW-IR	Shortwave Infrared Radiation
FTIR	Fourier transform infrared spectroscopy
DSC	Differential scanning calorimetry
TGA	Thermogravimetric analysis
SEM	Scanning electron microscope
S	Seconds
Hr	Hours
N	Newton

Other tests

Pull-Off Strength of Coatings Using Portable Adhesion Testers (ASTM D 4541)

Identifying testing standards for coatings are essential considering the nature of the bonding and the characteristics of the coating material. Apart from the ASTM D429, we also investigated the other standards. Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers (ASTM D 4541), and to a weekly bonded coatings, Standard Test Methods for Measuring Adhesion by Tape Test (ASTM D 3359) are more some testing standards.

In-house development of testing equipment adhering to the ASTM D4541 were carried out.

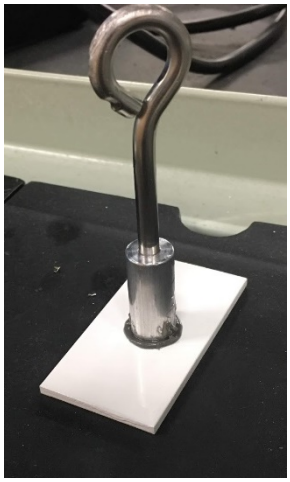

Fig. A1. a) Pull of rod b) Strongly glued to the top coat/substrate c) Clamped to the pull of device

Fig. A2. Hydraulic press

The hydraulic probe in the setup pushes the lever mechanism down thus pulling the pull rod up. The setup has the facility to give an analog reading as well as a digital reading through a connected computer. Time takes to glue the top surface to the top coat was considerably high and as a result this test was abandoned. To capture the peel strength reduction, it is very important to investigate the bond strength right after the IR treatment. There is no noticeable difference in bond strength when compare the treated sample after keeping it at room temperature for 30 minutes.

FTIR of Aluminum and composite base substrate

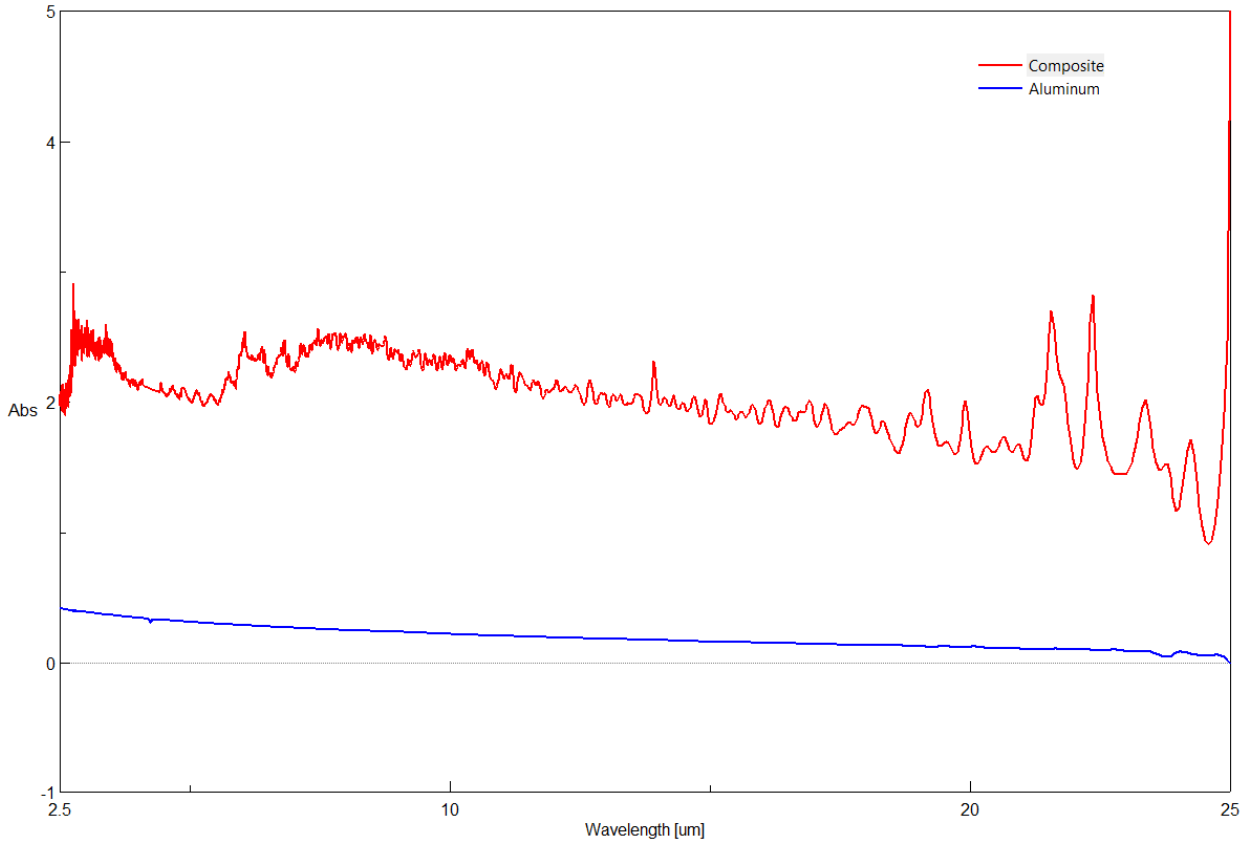

Fig. A3. % Absorption of IR spectra of composite and Aluminum base substrate material